Miscellaneous Paper EL-96-8
September 1996

**US Army Corps of Engineers**
Waterways Experiment Station

# An Evaluation of Solidification/Stabilization for Treatment of a Petroleum Hydrocarbon Contaminated Sludge From Fort Polk Army Installation, Louisiana

*by Michael G. Channell, Kurt T. Preston*





Prepared for U.S. Army Installation, Fort Polk

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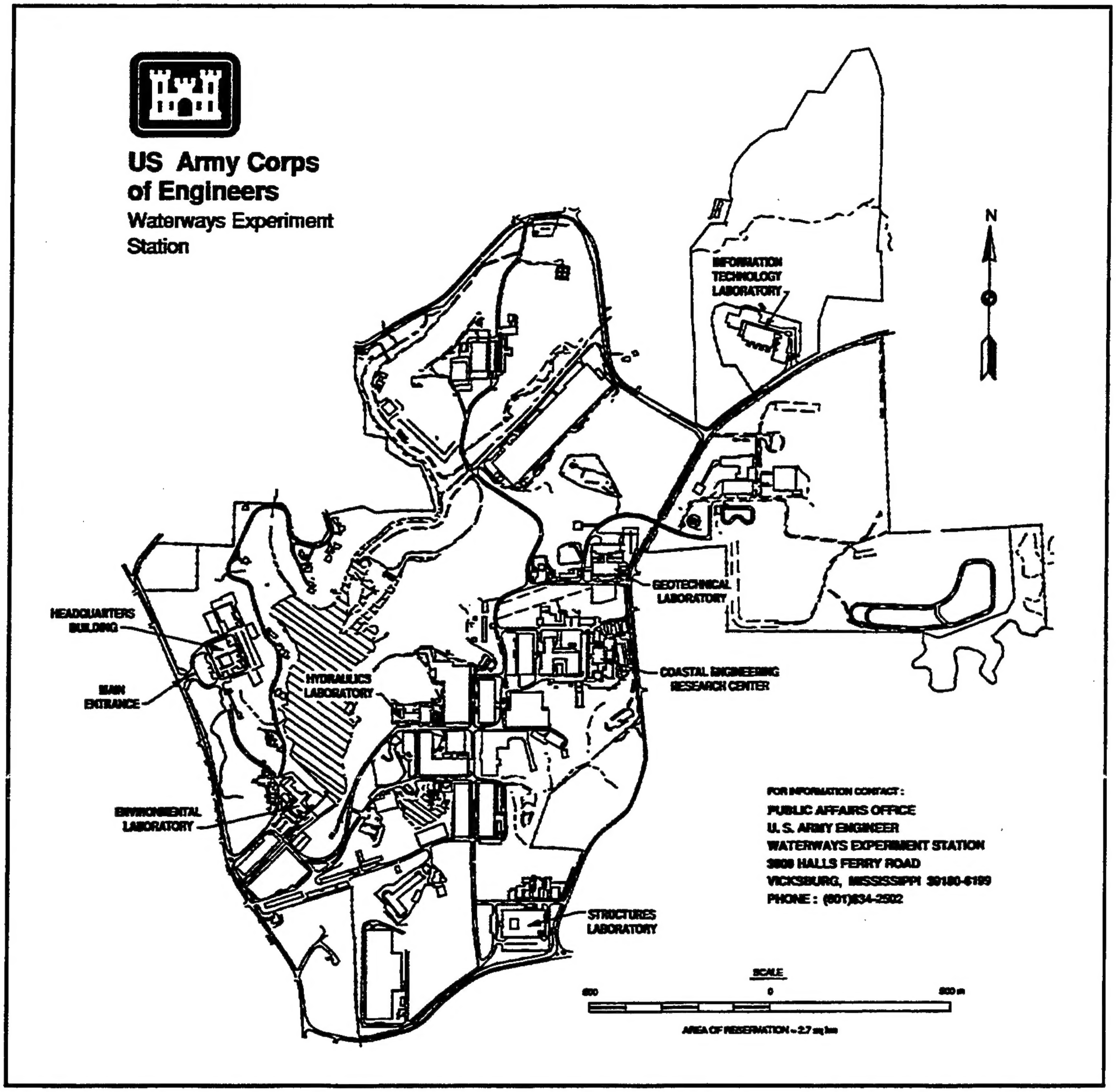
U.S. Army Corps of Engineers
Waterways Experiment Station
3909 Halls Ferry Road
Vicksburg, MS 39180-6199

Final report

Approved for public release; distribution is unlimited

Prepared for U.S. Army Installation, Fort Polk, LA




**Waterways Experiment Station Cataloging-in-Publication Data**

Channell, Michael G.
An evaluation of solidification/stabilization for treatment of a petroleum hydrocarbon contaminated sludge from Fort Polk Army Installation, Louisiana / by Michael G. Channell, Kurt T. Preston ; prepared for U.S. Army Installation, Fort Polk, LA.
47 p. : ill. ; 28 cm. — (Miscellaneous paper ; EL-96-8)
Includes bibliographic references.
1. Sewage sludge — Louisiana — Fort Polk — Evaluation. 2. Sewage sludge digestion —Louisiana — Fort Polk — Evaluation. 3. Silicates — Louisiana — Fort Polk — Testing. 4. Solidification — Evaluation — Testing. I. Preston, Kurt T. II. United States. Army. Corps of Engineers. III. U.S. Army Engineer Waterways Experiment Station. IV. Environmental Laboratory (U.S. Army Engineer Waterways Experiment Station) V. Fort Polk. VI. Title. VII. Series: Miscellaneous paper (U.S. Army Engineer Waterways Experiment Station) ; EL-96-8.
TA7 W34m no.EL-96-8

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# Preface

This report was prepared for Fort Polk Army Installation, Louisiana, by the U.S. Army Engineer Waterways Experiment Station (WES). The work was performed during the period 1 March to 15 June 1995 by Mr. Michael G. Channell and Dr. Kurt T. Preston of the Environmental Restoration Branch (ERB), Environmental Engineering Division (EED), Environmental Laboratory (EL), WES. Chemical analyses were performed by the Environmental Chemistry Branch (ECB). The work was conducted under the direct supervision of Mr. Daniel Averett, Chief, ERB, and under the general supervision of Mr. Norman R. Francingues, Chief, EED, and Dr. John W. Keeley, Director, EL. Project officer for the U.S. Army Fort Polk Installation was Dr. Charles Stagg.

At the time of publication of this report, Dr. Robert W. Whalin was Director of WES. COL Bruce K. Howard, EN, was Commander.

This report should be cited as follows:

> Channell, M. G., and Preston, K. T. (1996). "An evaluation of solidification/stabilization for treatment of a petroleum hydrocarbon contaminated sludge from Fort Polk Army Installation, Louisiana," Miscellaneous Paper EL-96-8, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

# Conversion Factors, Non-SI to SI Units of Measurement

Non-SI units of measurement used in this report can be converted to SI units as follows:

| Multiply | By | To Obtain |
|---|---|---|
| acres | 4,046.873 | square meters |
| cubic feet | 0.02831685 | cubic meters |
| degrees (angle) | 0.01745329 | radians |
| feet | 0.3048 | meters |
| gallons (U.S. liquid) | 3.785412 | liters |
| inches | 2.54 | centimeters |
| pounds (force) per square inch | 6.894757 | kilopascals |
| pounds (mass) | 0.4535924 | kilograms |
| square inches | 6.4516 | square centimeters |

# 1 Introduction

## Background

Fort Polk, Louisiana, established as a military training center in 1941, is a 198,325-acre[1] training installation located in the southwest portion of Louisiana near Leesville. The installation has been used continuously since 1941. Currently, Fort Polk is the home of the Joint Readiness Training Command (JRTC). The installation has a military population of 12,000 persons and a civilian population of 2,763 persons for a total population of 14,763. One of the major operations of the Fort Polk Army Installation is the operation and upkeep of many types of Army vehicles ranging from sedans and trucks to personnel carriers and tanks. The control of petroleum, oil, and lubricants (POL) associated with the operation and maintenance of these vehicles has changed greatly over the past 50 years. Whereas, 50 years ago the disposal of POL products was not of great concern, today it is. As a result of increased environmental concern, many Army installations are looking for ways to clean up past and present sites.

In the course of normal operations and training, soldiers and civilian personnel operate many vehicles on a day-to-day basis. Established operational maintenance procedures require vehicles to be free of dirt and cleaned before being parked at the various unit motor pools. Areas to wash the vehicles termed washracks have been constructed for this purpose. In most cases, vehicle washracks are simple concrete pads sloped to one side where the wash water is collected and drained into a gravity oil/water separator. The separators generally consist of a collection basin divided into two chambers. The first chamber acts as a settling tank, and the second chamber is designed to collect and retain floating oil. Water is passed through the oil/water separator to the installation sanitary sewer.

In January 1994, the installation environmental coordinator (IEC) contacted the U.S. Army Engineer Waterways Experiment Station (WES) for assistance in solving an environmental problem associated with managing the residues derived from the secondary settling basins of oil/water separators on the

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[1] A table of factors for converting non-SI units of measurement to SI units is presented on page vi.

installation. The installation manager described several major concerns related to the oil/water separator system including the presence of a large residual of petroleum product in the secondary chambers, the presence of solid debris in the separators, malfunctioning oil skimmers, and human operating errors.

Currently, the residues in the secondary chambers are allowed to build up. This is allowed to occur because it is unclear how to dispose of the residues in the most economic manner. During rain events, the oil/water separators function to some extent to keep the oils from reaching the installation wastewater treatment plant. However, the concern is that at some point the loading of the secondary chambers will overcome the capacity of the separators. The result will be a serious challenge to the installation wastewater treatment plant.

## Fast Track Solidification/Stabilization Efforts

A major concern associated with the residues was the likelihood that they might be considered a hazardous waste. POL products are not generally considered hazardous, except under specific State law, i.e., California, but the ease of access to the secondary chambers, the duration of their access (~25-30 years), and the varied military operations in the vicinity of the chambers caused concern. From the first, it was unclear which criteria would cause the materials to be hazardous, but the consensus was that heavy metals might be a problem due to vigorous cleaning performed on the vehicle engine areas. These assumptions proved themselves correct upon later investigation. However, at the initial meetings, the decision was made to invest in the investigation of a method to solidify a high oil sludge contaminated with heavy metals before it was verified that the condition actually existed. This decision was fortuitous. The only area in which the sludges were observed to be near or cross the threshold of being considered a hazardous waste was in the area of toxicity characteristic leaching procedure (TCLP) metals, particularly cadmium.

Once a determination is made of a hazardous waste under the Resource Conservation and Recovery Act (RCRA), as enacted through the Hazardous and Solid Waste Amendments of 1984 (HWSA), new responsibilities on the handlers of hazardous waste are incurred. In particular, HWSA prohibits the land disposal of untreated hazardous waste (RCRA sections 3004 (d)(1), (e)(1), (g)(5), 42 USC 6924 (d)(1), (e)(1), (g)(5)). Specific language under HSWA bans the land disposal of wastes containing free liquid in landfills. In addition, the utilization of adsorbents to remove free water is prohibited, and specifically stated is that materials used to treat free water must have evidence of a chemical reaction [(RCRA section 3004 (c)(1), USEPA 1982)]. As a result, a special issue is the disposal of liquid waste. In an effort to address the free liquids, the U.S. Environmental Protection Agency (USEPA) issued OSWER (the Office of Solid Waste and Emergency Response) Policy Directive 9487.00-2A (USEPA 1986a), which stipulates the development of an

Unconfined Compressive Strength (UCS) of 50 psi can be used as a measurement of meeting the chemical reaction, free liquid criteria.

The primary goal of this investigation was to meet the spirit of RCRA and to treat free liquids. Much of the experimental work performed for the establishment of these treatment standards in conjunction with solidification/stabilization (S/S) was conducted at WES under the direction of the USEPA's Office of Research and Development, and it was felt that this work might allow WES to continue to develop the technology. The general S/S protocol utilized for treatment standard development by USEPA was outlined in Bricka, Holmes, and Cullinane (1988). This protocol was utilized for this experimentation.

## Description

S/S is a process that involves the mixing of a contaminated soil, sludge, or liquid with a binder material to enhance the physical and chemical properties of the soil/sludge and to chemically bind any free liquid (USEPA 1986c). Solidification is generally conceptualized as the enhancement of the physical characteristics of the waste material. This is accomplished by reducing exposed surface area, which in turn lowers the convective transport of contaminants from the waste. Solidification usually entails the incorporation of the waste into a solid matrix or monolith. In comparison, stabilization involves the reaction of the waste's hazardous waste constituents with the S/S reagents to immobilize or otherwise contain them. The stabilization process may be as simple as the addition of lime or a sulfide source to a heavy metal liquid waste or may involve the development of special reagents specifically formulated to interact with the waste components. Most commercial vendors use a combination of solidification and stabilization to maximize the contaminant immobilization capability of the treated waste.

Several binder systems are currently available and widely used for the S/S of hazardous wastes (Cullinane, Jones, and Malone 1986). Typical binders include portland cements, pozzolans, and thermoplastics. Most common S/S techniques are designed with either portland cement or some type of pozzolan as the basic reagent. Portland cement is widely available, relatively economical, and well known to the general public as producing a very durable product. Pozzolans are siliceous materials that, when added to a source of lime, will go through a cementation process similar to portland cement but at a much slower rate. Fly ash and blast-furnace slag are common pozzolans that are generally considered waste materials themselves. Kiln dust is also a pozzolan and a waste material. Kiln dust is generated during production of lime or cement. Although the quality of kiln dust varies, kiln dust generally contains enough lime and fly ash to set with the addition of water.

In many cases, the S/S process is changed to accommodate specific contaminants and soil matrices. Generally, this is accomplished through the addition of admixtures. Soluble silicates, organophilic clays, activated carbon,

as well as a host of other organic and inorganic chemicals are routinely used as admixtures. For hazardous waste containing primarily metal contaminants, generally a cement or pozzolan binder makes up the bulk of the additive. Small quantities of admixture materials are added to the waste/binder mixture for a desired specific effect. Many of the proprietary processes marketed by the vendors of S/S are based upon admixtures.

Since it is not possible to consider all feasible modifications to an S/S process in this study, investigation of the S/S effectiveness was narrowed to focus only on generic process types (such as portland cement or lime/fly ash). The performance observed for a specific S/S system may vary widely from its generic type, but tailored processes generally are believed to perform equal to or better than the generic formulations. Typically, there is no need to evaluate proprietary S/S processes or admixtures if generic S/S processes prove sufficient to meet treatment goals. A comprehensive general discussion of admixtures and proprietary S/S processes is given in Malone and Jones (1979); Malone, Jones, and Larson (1980); and USEPA (1986c).

## Objective and Scope of Study

The objective of this effort was to develop a suitable solidification method for petroleum hydrocarbon contaminated washrack sludges for Fort Polk Army Installation. Specific goals included the following:

*a.* Determine if S/S techniques can be developed for the contaminated sludge collected from Fort Polk washracks.

*b.* If developed, evaluate the physical and chemical properties of the sludge to determine if the techniques will substantially reduce the amount of leachable contaminants and improve the physical handling properties of the sludge.

*c.* To generate physical and chemical data that can be used in the design specification and cost estimation of full-scale implementation.

## Organization of Report

This report is divided into four basic sections:

*a.* *Introduction.* Briefly describes the background for this study and introduces the concept of S/S.

*b.* *Materials and Methods.* Describes the methods used for sampling, treatment, and testing of the contaminated sludge.

*c.* *Discussion of Results*. Discusses results for the untreated sludge, initial screening test results, and detailed evaluation test results.

*d.* *Conclusions and Recommendations*. Based on the results of the testing program. In addition, the raw tabulated results for samples tested are presented in Appendixes A-C.

# 2 Materials and Methods

## General Approach to the Investigation

This investigation was conducted in the following five phases.

*a.* *Phase I: Sample Collection.* Contaminated sludge was collected from one washrack at Fort Polk Army Installation.

*b.* *Phase II: Homogenization.* The sludge was homogenized onsite when the samples were collected.

*c.* *Phase III: Preliminary Testing.* Tests were performed to determine the appropriate amount of binder and water to be added to the sludge for the detailed evaluation. Physical tests were performed on the samples to evaluate strength development properties for each mixture.

*d.* *Phase IV: Detailed Evaluation.* Based on the information from preliminary testing, samples were prepared for detailed evaluation. Physical tests and contaminant leach tests were performed on the samples to evaluate the effectiveness of S/S.

*e.* *Phase V: Data Analysis and Report Preparation.* Test data were consolidated and evaluated.

## Sample Collection

The material of interest was a contaminated sludge collected from one of approximately 50 washrack water collection tanks used at Fort Polk. A priori knowledge of the problem resulted in the selection of sludges from one particular settling basin at the installation. The basin was widely regarded by the Fort Polk environmental personnel as the most contaminated at the installation. Contaminants of interest were heavy metals and petroleum hydrocarbons. Two 55-gal drums of sludge were collected for the S/S study. Three distinct layers of sludges and oil-laden water were identified in the basin selected. The top layer was approximately 2 to 3 ft thick and very viscous.

This was the sludge collected for the S/S testing. The second layer was a flowable black liquid, and the third layer was clear liquid. The top layer of sludge was collected by using an ITT A-C pump to transfer the sludge from the washrack collection tank to 55-gal drums. Once the drums were filled, they were homogenized by using the pump to recirculate the sludge within each drum.

## Untreated Soil Characterization

### Chemical tests

**Bulk analysis.** The two drums (Replicate A and B) of the untreated sludge were subjected to chemical analysis for metals and total recoverable petroleum hydrocarbons (TRPHs) to determine the contaminant concentration in the sludge. Bulk chemical metal analyses were performed on the sludge prior to initiation of the characterization of the sludge.

**Toxicity characteristic leaching procedure.** The two sludge replicates were subjected to the TCLP extraction procedure to determine the hazardous characteristics and to measure the contaminant mobility as defined by the USEPA (USEPA 1986d). This method consisted of passing the sample through a 9.5-mm standard sieve. The sample was placed in a 0.5 N acetic acid extract or an acetate buffer extract, depending on the buffering capacity of the sludge, at a 20:1 liquid to solids ratio. The sludge and extract were tumbled end over end for 18 hr. At the completion of this period, the sample was filtered once using a Whatman GF/F 0.75-$\mu$m filter. The filtered extracts were placed in precleaned bottles and stored at 4 °C prior to analysis. Each extract was analyzed for metals and TRPHs.

### Physical tests

Physical characteristics of the untreated sludge were evaluated using the following test procedures. Test specimens were prepared in accordance with the requirements of the test method shown below.

**Moisture content.** The moisture content was determined for each of the two replicates in accordance with a modified American Society of Testing and Materials (ASTM) method D-2216 (ASTM 1992a). This method was modified by drying the sample to a constant weight at 60 °C. Lower temperatures are used to avoid removing volatilization of large volumes of the contaminants and to reduce the release of hydrated water. The moisture content measurements were used to calculate the dry weight of each sample.

**Bulk density.** In the initial tests, the bulk density for each of the two replicates as determined in accordance with American Society of Agronomy (AA) Method 13 (AA 1965). This test was performed on the untreated samples by loosely placing a known mass of sludge into a mold of known volume.

This density represents the uncompacted laboratory density of the sludge as used in the S/S treatability studies. The laboratory bulk density is not the in situ density, which is measured in the field. The bulk densities were calculated using the mass and volume data and were reported in units of pounds per cubic foot. In the final tests, bulk density determinations were performed in triplicate for each binder, each formulation, and each replicate. A total of 18 cubes were tested for the sludge for bulk density after they had cured for 28 days under a controlled environment.

**Unconfined compressive strength.** The unconfined compressive strength (UCS) was determined for the two replicates of the sludge. The UCS measurements were conducted according to ASTM C-109 (ASTM 1992b). The samples were aged for 7 days in an environment controlled at 23 °C ± 2 °C and 95 percent ± 5-percent relative humidity prior to testing. After removal from the mold, the surface area of each sample was determined using a Fowler Max-Cal Caliper. The cubes were placed in plastic bags; while in the bag, each cube was subjected to a compressive force until the cube fractured. A Tinius Olsen Super-L compressive apparatus was used to supply this force and indicate the compressive strength at which the cubes fractured. The UCS of each cube was reported as the force required to fracture the cube per square inch of surface area (pounds per square inch). UCS testing was performed on both the initial and detailed evaluations.

**Resistance to penetration.** The Cone Index (CI) determination was performed on each replicate of the sludge and was conducted according to TM 5-540 (Headquarters, Department of the Army (HDQA) 1971). The CI measures the resistance of a material to the penetration of a 30-deg right circular cone. The CI value is reported as force per unit surface area (pounds per square inch) of the cone base required to push the cone through a test material at a rate of 72 in./min. Two cones are available for this test: (a) the standard WES cone having an area of 0.5 sq in. and (2) the airfield penetrometer having a base area of 0.2 sq in. Because of its smaller cone, the airfield penetrometer can measure larger CI values. It was convenient to use the standard WES cone penetrometer on materials with a CI up to 300 psi. The maximum CI value that can be measured by the airfield penetrometer is 750 psi; therefore, materials having CI values greater than 750 psi are reported simply as >750 psi.

**Bleed water.** Bleed water is defined as the relative quantity of mixing water that will bleed from a freshly mixed concrete. The amount of bleed water produced was determined for the detailed evaluations only. Each formulation selected for detailed evaluation was measured using ASTM Method C 232 (Bleeding of Concrete, ASTM 1987). To determine if the mixtures produced bleed water, samples were visually inspected to determine if a water layer was detected. ASTM method C-232 method A was used to measure the quantity of this bleed water.

**Cracking.** There are no known standard test procedures for measuring the degree of cracking. In the detailed examinations, the sample specimens were

visually inspected for cracks. Development of cracks is considered to be detrimental to solidified samples. The formation of cracks increases the surface area of the sample. One of the purposes of the S/S process is to decrease the surface area of the waste by the formation of a monolith. The formation of cracks increases the potential for water infiltration by increasing the waste's surface area, thus increasing the potential for contaminant leaching.

**Set time.** The set time is defined as the time required to develop sufficient rigidity following mixing to resist the penetration of a standard rod or needle. Set time for the detailed evaluations were evaluated using the CI as described above. Measurements were taken on samples after they had cured 2, 4, 8, 24, and 48 hr. CI tests were performed in triplicate for each binder, each formulation, and each replicate.

## Preparation of the Test Specimens

Two processes were used to solidify/stabilize the sludge from the washrack collection tank and were differentiated by the type of binder material used in the process. The two processes used for this study were portland cement and portland cement with added class F fly ash. A compositional and chemical analysis of binders used in this study is presented in Tables 1 and 2. The S/S process involves the addition of water and binder material to the waste followed by a mixing and a curing period. A schematic flowchart of S/S processing is shown in Figure 1.

The initial screening test (IST) is used to narrow the range of binder-to-soil ratios (BSRs) and water-to-soil ratios (WSRs) necessary for evaluation of the material during the detailed evaluation portion of the study. The sludge collected from the washracks contained water in amounts sufficient for testing without further addition. For the IST, varying ratios of binders were added to the sludge to evaluate the physical characteristics of the solidified sludge. The initial waste/binder screening test involved mixing binder and sludge in a K455S Hobart mixer for 10 min. A total of two binder and four BSRs were evaluated for the sludge in the IST phase of the study. Upon completion of the two binder and four BSR trials, poor results dictated a new approach. It was decided to try the addition of an oil absorbent, diacalcium silicate, and an addition of an organophilic clay to bind the petroleum hydrocarbons and then to add the binder to the mixture. Four ratios of the dicalcium silicate and one BSR of cement was chosen for this phase of the IST. Three ratios of the organophilic clay and one BSR of cement was chosen for testing of this phase of the IST.

After each formulation was mixed, it was placed in a 4-in.-diam by 4-in.-high cylindrical plastic mold. These mixtures were either poured into the molds and vibrated on a Syntron model VP61D1 vibration table or compacted in the molds using the standard Proctor density ASTM D-698 (ASTM 1992a). The samples were placed in a controlled environment at 23 °C ± 2 °C and 95-percent relative humidity ± 5 percent until needed for testing.

**Table 1**
**Compositional Analysis of Binders**

| Compositional Analysis | Cement Type I, Percent | Fly Ash Class F, Percent |
|---|---|---|
| $SiO_2$ | 20.47 | 49.67 |
| $Al_2O_3$ | 5.40 | 29.15 |
| $Fe_2O_3$ | 3.58 | 7.11 |
| CaO | 64.77 | 1.26 |
| MgO | 0.87 | 1.43 |
| $SO_3$ | 2.73 | 0.23 |
| Insoluble residue | 0.17 | 70.70[1] |
| Moisture loss | 0.43 | 0.12[2] |
| Loss on ignition | 0.96 | 4.07 |
| $TiO_3$ | 0.28 | 0.20 |
| $Mn_2O_3$ | 0.06 | 0.00 |
| $P_2O_5$ | 0.28 | 1.00 |
| **Total Alkali** | | |
| $Na_2O$ | 0.12 | 0.23 |
| $K_2O$ | 0.28 | 2.33 |
| Na | 0.05 | 0.10 |
| K | 0.11 | 0.97 |
| Total as $Na_2O$ | 0.30 | 1.76 |
| **Acid Soluble Alkali** | | |
| $Na_2O$ | 0.12 | 0.06 |
| $K_2O$ | 0.28 | 0.50 |
| Na | 0.05 | 0.03 |
| K | 0.11 | 0.21 |
| **Water Soluble Alkali** | | |
| $Na_2O$ | 0.018 | 0.050 |
| $K_2O$ | 0.139 | 0.105 |
| Na | 0.0075 | 0.0210 |
| K | 0.0577 | 0.0440 |

[1] Insoluble residue includes $SiO_2$.
[2] Free water.

**Table 2**
**Chemical Analysis of Binders**

| Chemical Analysis | Cement Type I, mg/kg | Fly Ash Class F, mg/kg |
|---|---|---|
| Si | 95,700 | 32,400 |
| S (total) | 10,800 | 31,200 |
| Ti | 1,400 | 600 |
| P | 900 | 200 |
| Sb | <1.77 | 13.3 |
| As | 13.1 | 172 |
| Be | 2.13 | 28.9 |
| Cd | 0.284 | 1.01 |
| Cr | 61.3 | 139 |
| Cu | 14.9 | 196 |
| Pb | 2.13 | 57.7 |
| Hg | <0.100 | <0.100 |
| Ni | 25.9 | 190 |
| Se | <17.7 | <19.5 |
| Ag | <3.54 | <3.90 |
| Tl | <10.6 | 13.6 |
| Zn | 41.8 | 211 |
| Al | 23,100 | 150,000 |
| Ba | 178 | 1,350 |
| Ca | 454,000 | 12,000 |
| Cd | 10.6 | 77.2 |
| Fe | 25,400 | 50,700 |
| Mg | 5,460 | 6,040 |
| Mn | 503 | 156 |
| Na | 1,270 | 2,740 |
| Sn | 195 | 118 |
| V | 55.6 | 351 |

Determination of the optimal BSRs and absorbent-to-sludge ratios was based on the results of the CI test performed on the IST samples during a 48-hr curing period. CI measurements, as described in the sludge characterization, were performed on these samples at 2, 4, 8, 24, and 48 hr after curing.

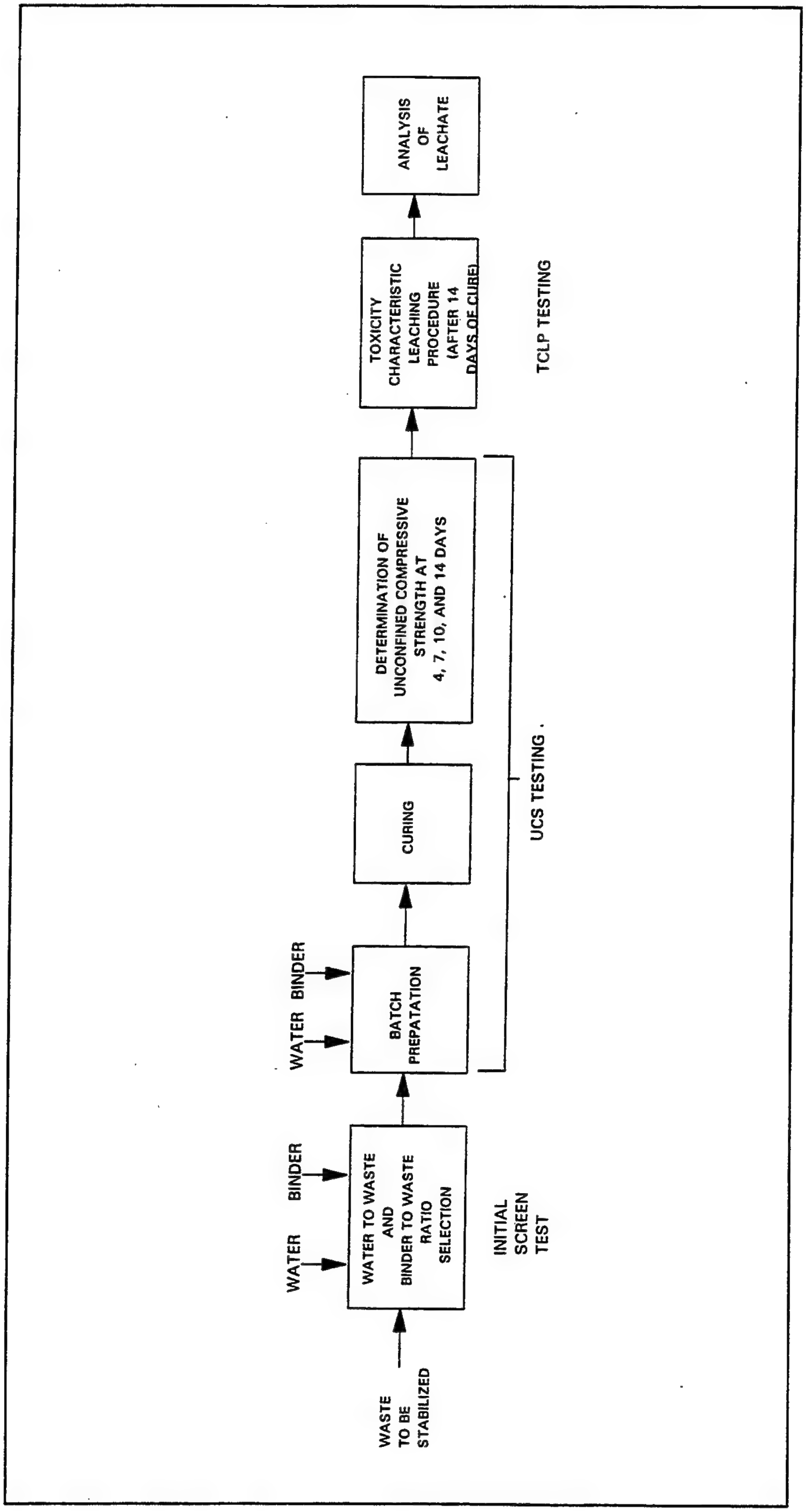


Figure 1. Schematic flowchart for stabilization processing

## Detailed Evaluation Testing

Based on the results of the initial screening test, one absorbent, dicalcium silicate, in the ratio of 1.35 was chosen for the detailed evaluation portion of this study. Four BSRs for cement and cement/fly ash were prepared in duplicate for the sludge for the detailed S/S evaluation. Solidified/stabilized specimens were prepared by mixing absorbent and binder with the contaminated sludge in a Hobart C-600 mixer.

The mixing procedure was as follows: 3,000 g of sludge and 4,050 g of dicalcium silicate were mixed for 5 min, after which the sides of the container were scraped to remove material adhering to the sides of the container. After the dicalcium silicate and sludge were mixed, the binder ratios of 0.5, 0.7, 0.9, and 1.1 cement and 0.3/0.3, 0.3/0.4, 0.4/0.3, and 0.4/0.4 cement/fly ash were added to the mixture; this mixture was mixed for an additional 5 min. When mixing was complete, the specimens were poured into molds. A variety of specimens were prepared for the various test protocols. To aid in removing test specimens from molds, a light coat of grease was applied to the molds used to cast the UCS specimens. Specimens used for the TCLP were placed in ungreased molds. Ungreased molds were used for the chemical tests to avoid possible chemical contamination from the grease. Immediately after the absorbent/binder/sludge mixtures were placed in the molds, they were vibrated on a Sentron model VP61D1 vibration table to remove voids. Since some of the mixtures were viscous, vibration was an ineffective method for removing voids. These specimens were tamped according to ASTM C 109-86 (ASTM 1987) using a model CT-25A tamper.

The molded solidified/stabilized materials were cured in the molds at 23 °C and 98-percent relative humidity for a minimum of 24 hr. Specimens were removed from the molds when they were observed to have developed sufficient strength to be free standing. The decision to remove the molds were professional judgements made by visual and tactile observation. The free standing monoliths were then cured under the same temperature and relative humidity conditions until required for further testing. In the detailed evaluations, the physical and chemical properties of the sludge were determined after the solidified/stabilized sludge had cured for 28 days.

# 3 Results of Contaminant Mobility and Physical Testing

## Untreated Sludge Testing

The untreated sludge was subjected to a battery of physical and chemical tests. The results of the physical tests are summarized in Table 3. The raw physical test results for the untreated sludge characterization are presented in Appendix A, Table A1. The purpose of this initial characterization is two-fold. First, engineering properties of the sludge were measured to provide data that describe the sludge; secondly, baseline data are collected for the untreated sludge to provide a basis of comparison for the various treatments applied.

It should be noted that the UCS for the sludge could not be performed (Table 3). Since the sludge contained water and oil, the molded specimens did not achieve physical strengthening and could not be removed from the molds except as a fluid. Because of this, the reported UCS for the sludge is zero. This UCS falls below the USEPA-recommended 50-psi criteria (USEPA 1986a).

**Table 3**
**Average Results of Physical Tests Conducted on Untreated Fort Polk Sludge**

| Replicate | Moisture Content Percent | Bulk Density $lb/ft^3$ | UCS, psi | Cone Index psi |
|---|---|---|---|---|
| A | 21 | 37.8 | 0 | 0 |
| B | 23 | 34.1 | 0 | 0 |

## Bulk Chemistry

The untreated sludge was analyzed to determine the total concentration of contaminants of concern. Table 4 presents the average results of the bulk chemical analyses of the Fort Polk washrack sludge. The raw data are presented in Appendix A, Table A2. Metals were present in the sludge at very low levels. TRPHs were present at extremely high concentrations of 31.4 percent of the total mass of the sample. With water and oil constituting approximately 53 percent of the total mass of the sludge, it is suspected that the remaining sludge is primarily made up of clay particles that are removed from the vehicles at the washracks. The oils are absorbed onto the clays, and thus this is the cause of the sludge floating on top of the other layers of liquid present in the washrack water containers.

**Table 4**
**Average Results of Bulk Chemistry for Untreated Fort Polk Washrack Sludge**

| Analyte | Concentration, mg/kg |
|---|---|
| Arsenic | <0.20 |
| Barium | 13.8 |
| Cadmium | 0.73 |
| Chromium | 0.885 |
| Lead | 3.29 |
| Mercury | <0.100 |
| Selenium | <0.20 |
| Silver | 0.1 |
| TRPH | 314,000 |

TCLPs were run in duplicate (Replicates A and B) for the untreated sludge. Metals analyses for the TCLP extracts indicated that all metals were below the reported detection limits. This was expected because of the low bulk concentration of the metals present in the sludge.

## Initial Screening Test Results

The results of the CI for the IST are presented in Figures 2, 3, and 4 for the cement binder, the dicalcium silicate/cement, and clay/cement, respectively. The raw IST data for the CI tests and the Material Safety Data Sheet for the absorbants are presented in Appendix B.

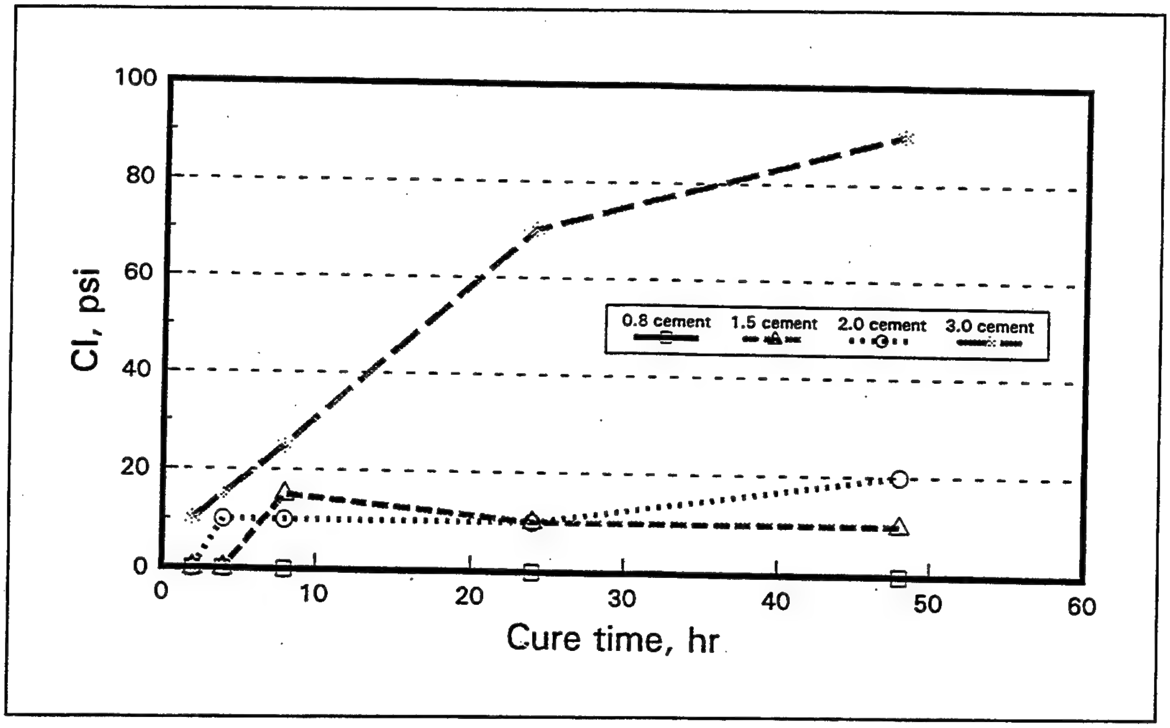


Figure 2. Results of CI for IST using cement binder

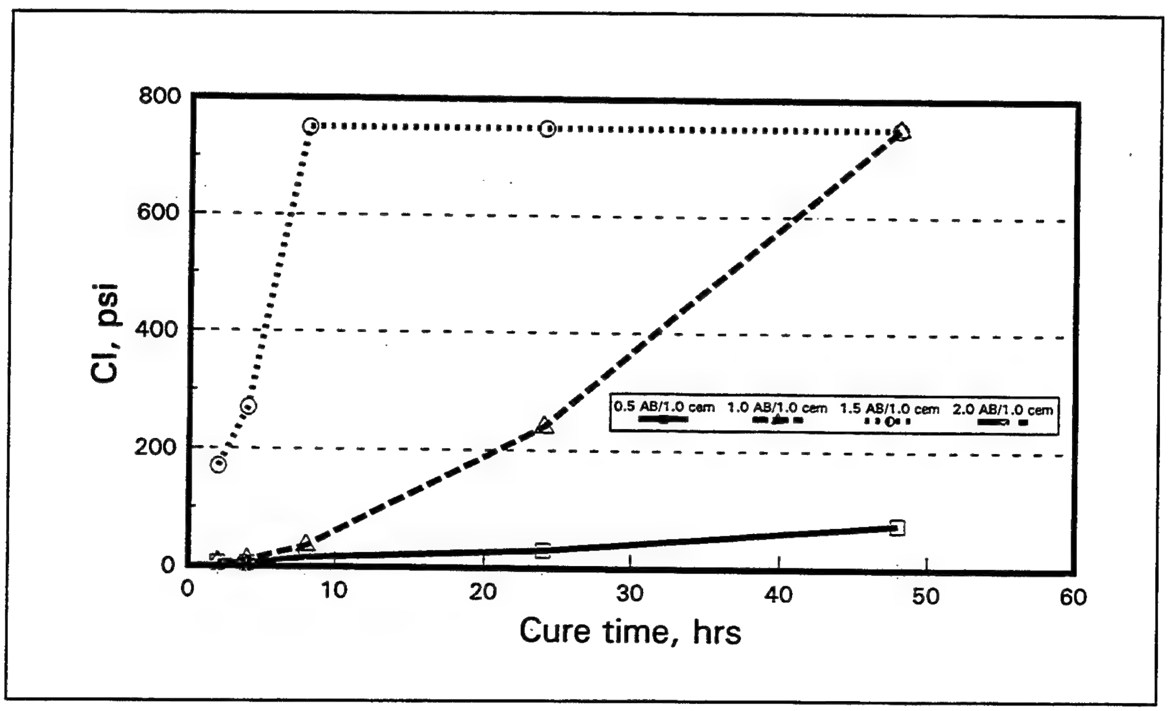


Figure 3. Results of CI for IST using dicalcium silicate/cement binder

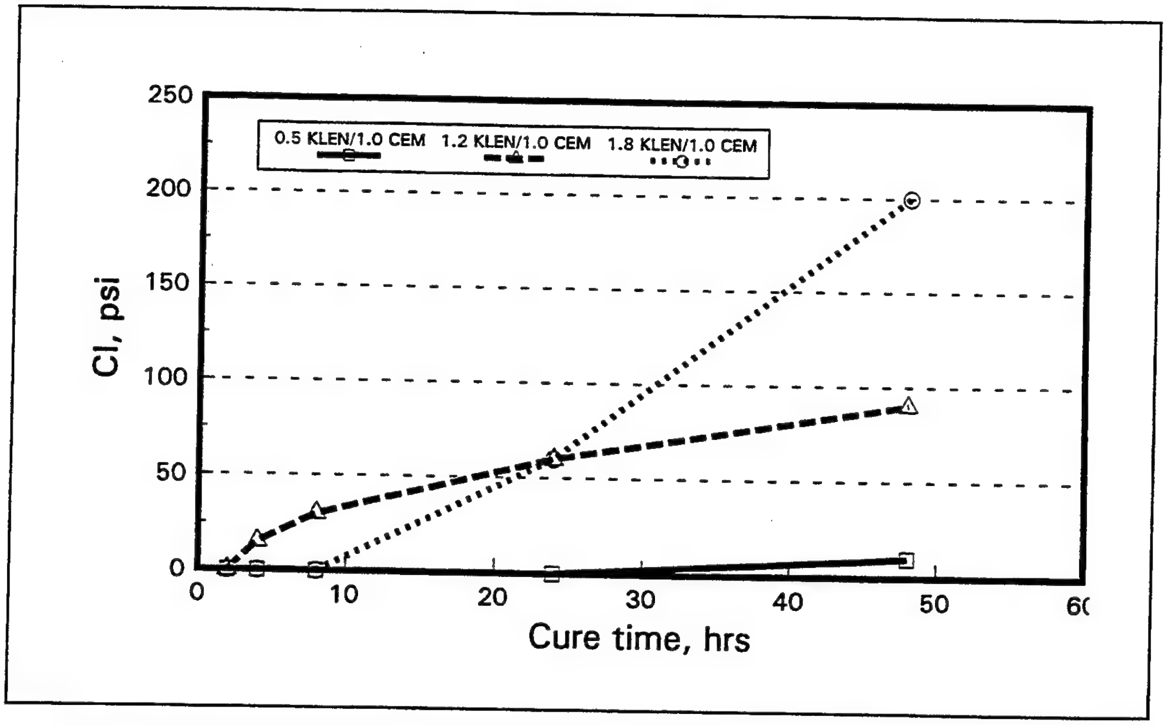


Figure 4. Results of CI for IST using organophilic clay/cement binder

Figures 2, 3, and 4 are plots of cure time versus CI for all sludge and binders evaluated in the IST. Figure 2 indicates that when cement alone is used for binding of the sludge, very little strength is gained by the sample. The highest cement ratio of 3.0 (300 percent by wet weight) gained the most strength of the samples tested, but the CI was below 100 psi after 48 hr of cure. Petroleum hydrocarbons have been demonstrated to have an adverse effect on the set of solidified/stabilized samples (Bricka and Jones 1993). This adverse effect is demonstrated by the results presented in Figure 2. During the addition of the cement binder, it should be noted that at the low BSRs, free water was noticed on the top of the mixture. This can be attributed to the phenomena of petroleum hydrocarbons coating the cement particles, thus preventing water from hydrating the cement particles. The absence of hydration results in excess water in the mixture and the observed water pooling on the top of the mixture. The presence of this free water was the element which forced the decision to react the oil with commercially available products and then add the cement to hydrate with the free water.

Figure 3 presents the results of the CI for IST using cement and dicalcium silicate. The ratio of 1.5 dicalcium silicate/1.0 cement performed the best for the ratios tested. This ratio rapidly gained strength and achieved the maximum, 750 psi, CI at 8 hr of cure. The 1.0 dicalcium silicate/1.0 cement gained strength throughout the 48 hr of testing achieving the maximum CI of 750 psi after 48 hr of cure. It should be noted that the 2.0 dicalcium silicate/ 1.0 cement did not gain any strength during the CI test. This is due to the

fact that the mixture was extremely dry and crumbled when subjected to the cone penetrometer.

Figure 4 presents the results of the CI for the IST using cement and the organophilic clay (Klensorb). The addition of the organophilic clay did improve the strength of the samples when compared with the samples prepared using only cement. Although the strengths did improve, they were still low with the 1.8 organophilic clay/1.0 cement ratio gaining the most strength with a 48-hr CI of 200 psi. The addition of the organophilic clay to the sludge did not perform as well as the samples prepared using dicalcium silicate for the CI test.

The CI results for the absorbent/cement binder ratios indicate that the maximum CI was produced from the samples prepared using the dicalcium silicate/cement binder ratios. Based on the results of the IST, a ratio of 1.35 dicalcium silicate with varying ratios of cement and cement/fly ash was selected for further evaluation.

## Detailed Evaluation Results

Based on the results of the ISTs, the ratio of 1.35 dicalcium silicate was selected for the detailed evaluation. Four ratios of cement and four ratios of cement/fly ash were selected for addition to the 1.35 dicalcium silicate/sludge for preparation of samples for the detailed evaluation.

A combination of five tests were used to measure the physical properties of the tested sludge. These tests included bulk density, bleed water, cracking, UCS, and CI. The raw data generated from these tests are presented in Appendix C, and the results for each test are discussed below.

### Bulk density

Figure 5 presents the average bulk densities for the 1.35 dicalcium silicate/cement binders compared with the untreated sludge bulk density. As shown in Figure 5, the treated samples more than doubled the bulk density of the untreated material. This is expected since absorbent and high ratios of cement were added to the sludge for solidification.

Figure 6 presents the average bulk densities for the 1.35 dicalcium silicate/cement/fly ash binders compared with the untreated sludge bulk density. All of the treated sample bulk densities were higher than the untreated sludge bulk density. Although the bulk densities were higher, the cement/fly ash binder samples had lower bulk densities than the cement binder samples. This is due to the fact that lower cement ratios were used for these samples and that the type F fly ash has a low bulking effect.

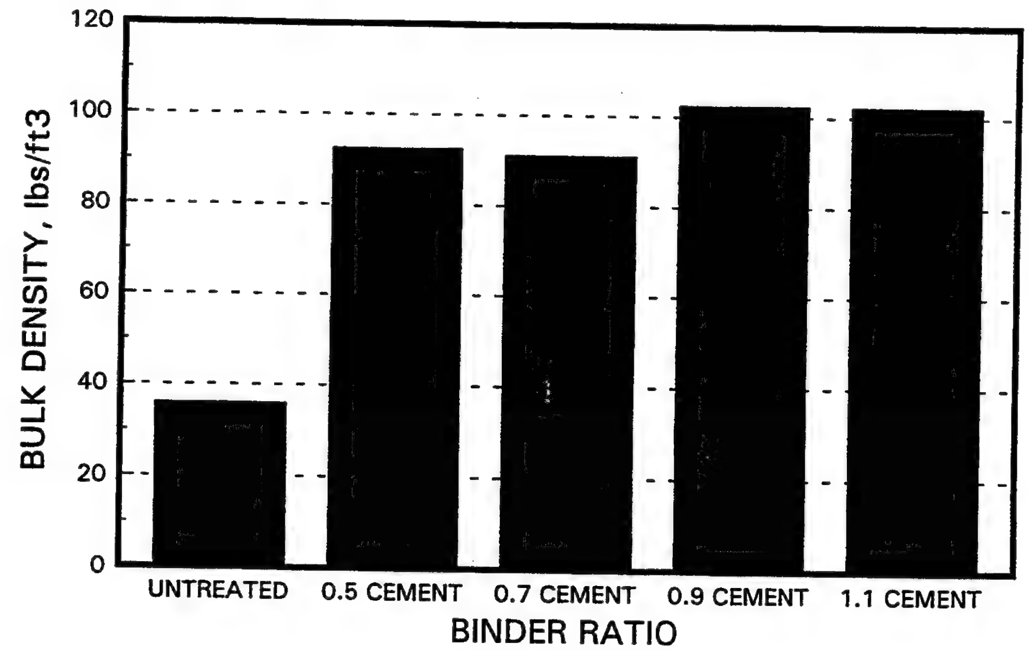


Figure 5. Average 28-day bulk density for samples using cement binder

### Bleed water and cracking

All of the specimens prepared were visually inspected for bleed water and cracking as described in the Materials and Methods section of this report. None of the samples prepared produced bleed water, and all samples were free from visual cracks. This is important because the generation of a large number of cracks could potentially increase the rate of leaching of the contaminant from the solidified sludge. The development of a large number of cracks could be an indication of incompatibility with the sludge and binder material. Although none of the samples indicated the presence of free water after the mixing was complete, it should be noted that after the 5 min of mixing of the sludge and dicalcium silicate, water was observed to form on top of the mixture. Once the binder agent was added to the mixture and mixed for 5 min, the free water was not present. It is apparent that this water was used for the hydration of the binder.

### Cone Index

Results of the CI test for the detailed evaluation are presented in Appendix C, Tables C1 and C2. Figures 7 and 8 present the average CI results for the samples solidified using the cement and cement/fly ash binders,

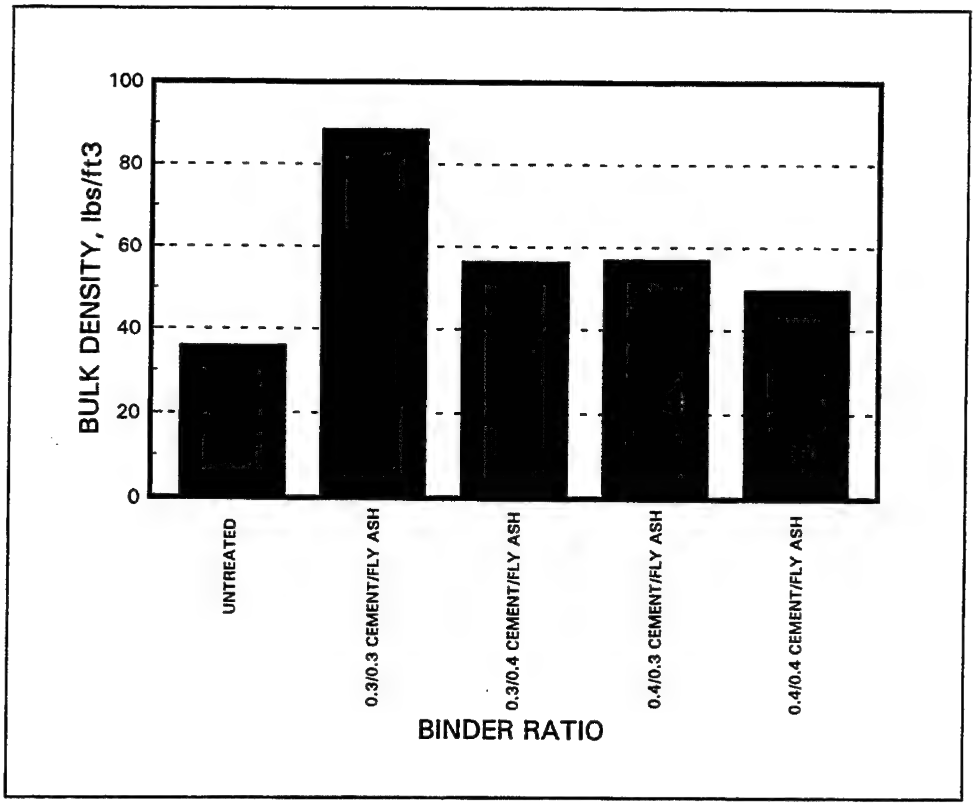


Figure 6. Average 28-day bulk density for samples using cement/fly ash binder

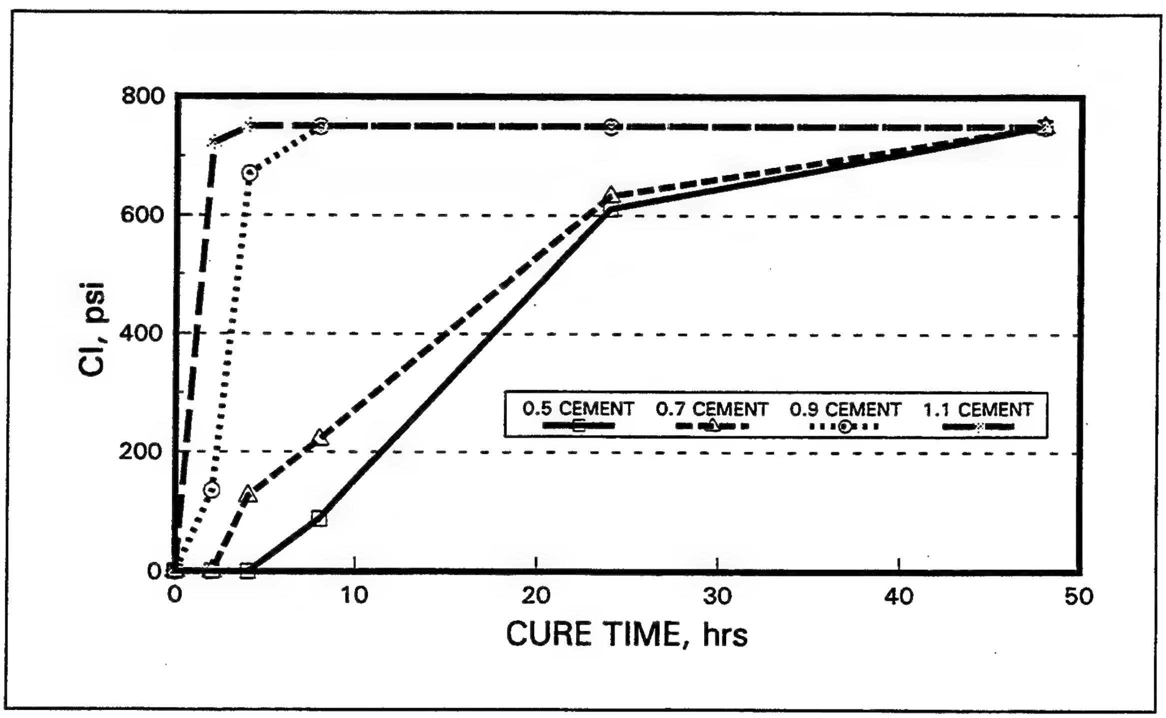


Figure 7. Average results for CI for detailed evaluation cement binder samples

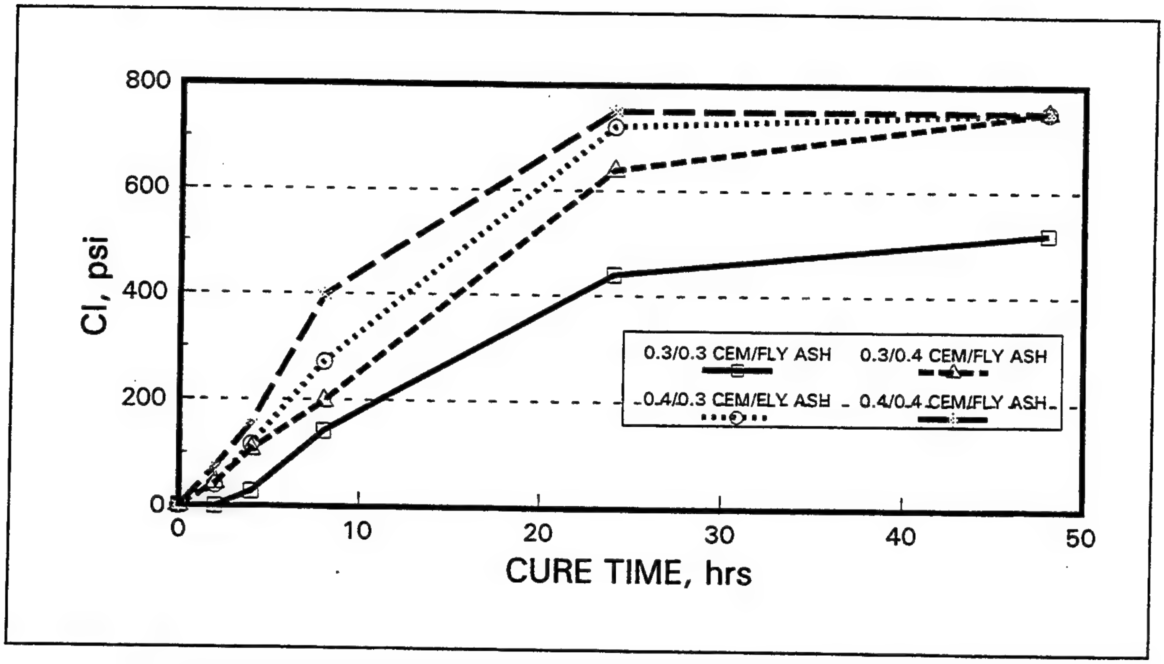


Figure 8. Average results for CI for detailed evaluation cement/fly ash samples

respectively. The data are averaged for the replicate samples (A and B) and are presented as the CI value (reported as pounds per square inch) versus the cure time in hours.

Figure 7 indicates that all of the samples for the cement binder ratios achieved the maximum CI of 750 psi after 48 hr of cure. Figure 7 shows that as the amount of binder is increased for the sludge/dicalcium silicate mixture, the samples develop strength more rapidly. The 0.5 and 0.7 cement BSR achieved a CI of 750 psi at 48 hr of cure. The 0.9 cement BSR achieved a CI of 750 psi at 8 hr of cure, while the 1.1 cement BSR achieved a CI of 750 psi at only 4 hr of cure. It should be noted that the 0.9 and 1.1 cement BSR samples did not vibrate down in the molds very well when placed on the vibration table. These samples had to be tamped into the molds to remove any voids that might form in the samples.

Figure 8 shows that all samples tested achieved a maximum of CI of 750 psi after 48 hr of cure except the 0.3/0.3 cement/fly ash BSR. The BSR of 04./0.4 cement/fly ash achieved the maximum CI of 750 psi after 24 hr of cure. Figure 8 shows that as more cement is added to the sludge/dicalcium silicate, the samples gain strength more rapidly. Also, the addition of fly ash to the mixture aids in the strength developed by the sample.

**USC.** Results of the UCS test for the detailed evaluation portion of this study are presented in Appendix C, Table C3. The data were averaged for the replicate samples (A and B), and these results are presented in Figures 9 and 10, as the UCS (reported in pounds per square inch) versus the cure time in hours.

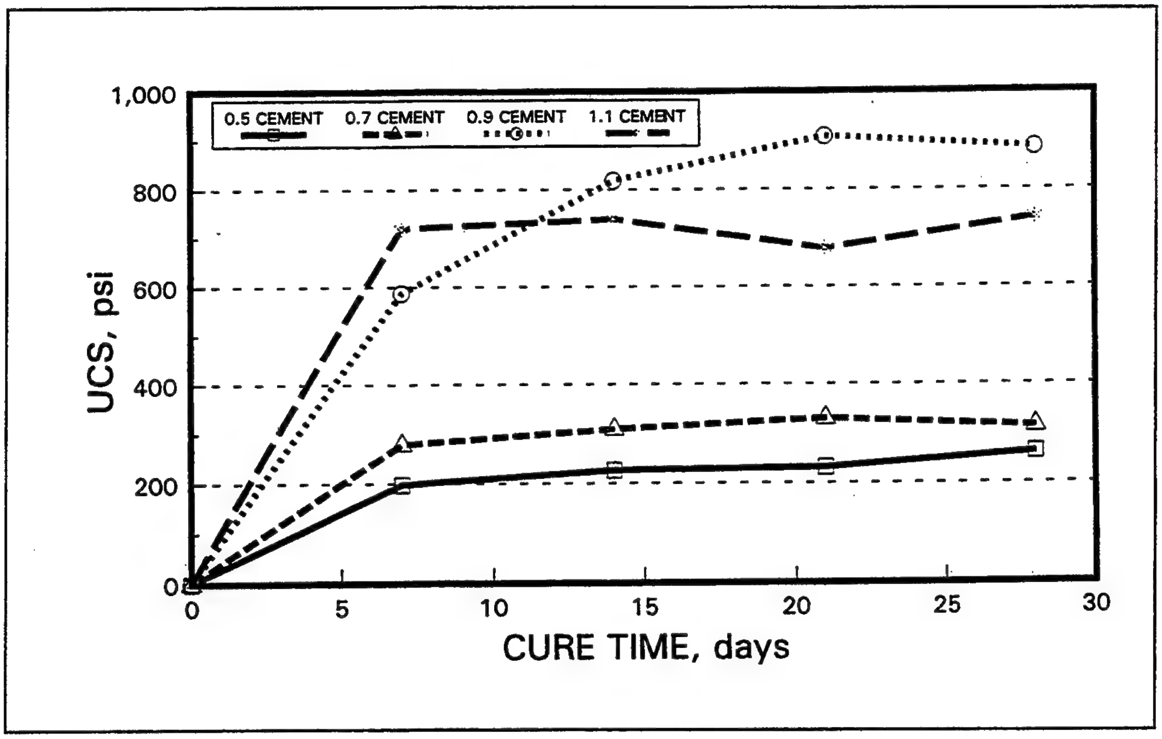


Figure 9. Average results for UCS for detailed evaluation cement samples

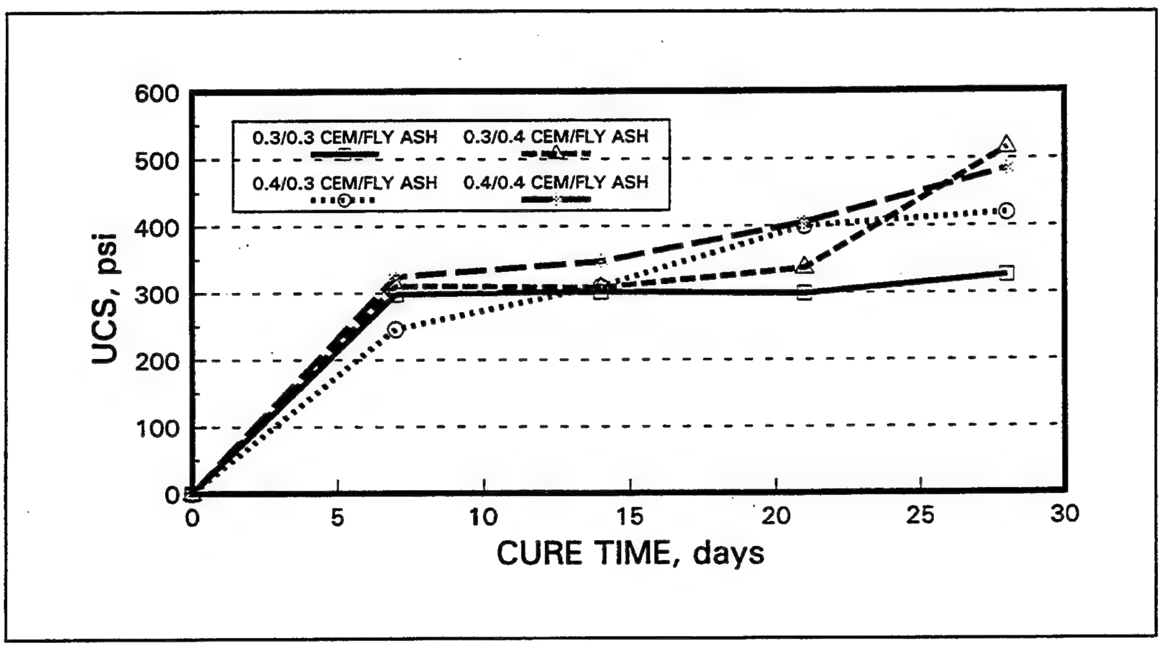


Figure 10. Average results for UCS for detailed evaluation cement/fly ash samples

Figure 9 presents the data for the UCS test performed on the cement BSRs. The sample prepared using the 0.9 cement BSR achieved the highest UCS of all the samples tested. All BSRs tested except the 0.9 BSR appeared to

achieve their ultimate strength at 7 days of cure. The UCS for these samples remained approximately the same for the remainder of the test time. The 0.9 BSR gained strength throughout the test time until Day 21, when it obtained its ultimate strength. All samples prepared using the cement BSRs exceeded the EPA criteria of 50 psi for the UCS test.

Figure 10 presents the data for the UCS test performed on the cement/fly ash BSRs. All samples prepared using the cement/fly ash BSRs gained strength throughout the cure time except for the 0.3/0.3 cement/fly ash BSR. The 0.3/0.3 cement/fly ash BSR achieved its ultimate strength at 7 days of cure and remained approximately constant for the remainder of the 28-day testing period. The samples prepared using the 0.4 fly ash binder achieved a greater strength than the samples prepared using the 0.3 fly ash binder. Although the cement/fly ash BSR samples did not gain as much strength as the cement BSR samples tested, the cement/fly ash BSR samples exceeded the EPA criteria of 50 psi for the UCS test.

## Contaminant Release Testing

Table 5 presents the average cement and cement/fly ash BSR results for metals and TCLP extract used in the detailed evaluation. The raw data are provided in Appendix C, Table C4. As expected, due to the low concentration of metals found in the untreated sludge, arsenic, cadmium, lead, mercury, and selenium were at or below current detection limits. Barium and chromium were detected, but were well below the TCLP regulatory limits of 100 and 5, respectively. From Table 5, it can be seen that metals found in the TCLP leachate do not pose a threat of leaching from the solidified samples.

**Table 5**
**Average Results of Metals for the TCLP, mg/ℓ**

| BSR | As | Ba | Cd | Cr | Pb | Hg | Se | Ag |
|---|---|---|---|---|---|---|---|---|
| **Cement Binder** | | | | | | | | |
| 0.5 | 0.0025 | 0.667 | <0.002 | 0.0393 | <0.002 | <0.002 | 0.003 | <0.01 |
| 0.7 | 0.0025 | 1.175 | <0.002 | 0.0376 | 0.002 | <0.002 | <0.002 | <0.01 |
| 0.9 | 0.0035 | 1.45 | <0.002 | 0.0278 | 0.0035 | <0.002 | <0.002 | <0.01 |
| 1.1 | 0.0025 | 1.47 | <0.002 | 0.0325 | <0.002 | <0.002 | <0.002 | <0.01 |
| **Cement/Fly Ash Binder** | | | | | | | | |
| 0.3/0.3 | <0.002 | 1.41 | <0.002 | 0.0313 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.3/0.4 | <0.002 | 1.195 | <0.002 | 0.0381 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.3 | <0.002 | 1.285 | <0.002 | 0.0280 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.4 | <0.002 | 1.205 | <0.002 | 0.0304 | <0.002 | <0.002 | <0.002 | <0.01 |

Figure 11 presents the average results for TCLP extract TRPHs for the cement and cement/fly ash BSRs used in the detailed evaluation. The raw data are provided in Appendix C, Table C5. The cement BSRs of 0.5 and 1.1 had the lowest concentration of TRPH for the cement BSRs tested with a concentration of 0.6 and 0.8 mg/ℓ, respectively. The cement BSRs of 0.7 and 0.9 had higher concentrations of TRPH with concentrations of 3.4 and 4.8 mg/ℓ, respectively. The cement/fly ash BSRs tested had TRPH concentrations between 0.6 and 1.1 mg/ℓ. Figure 11 indicates that TRPH concentration is lower in the cement/fly ash BSRs than the cement BSRs. This was an indicator of superior treatment performance.

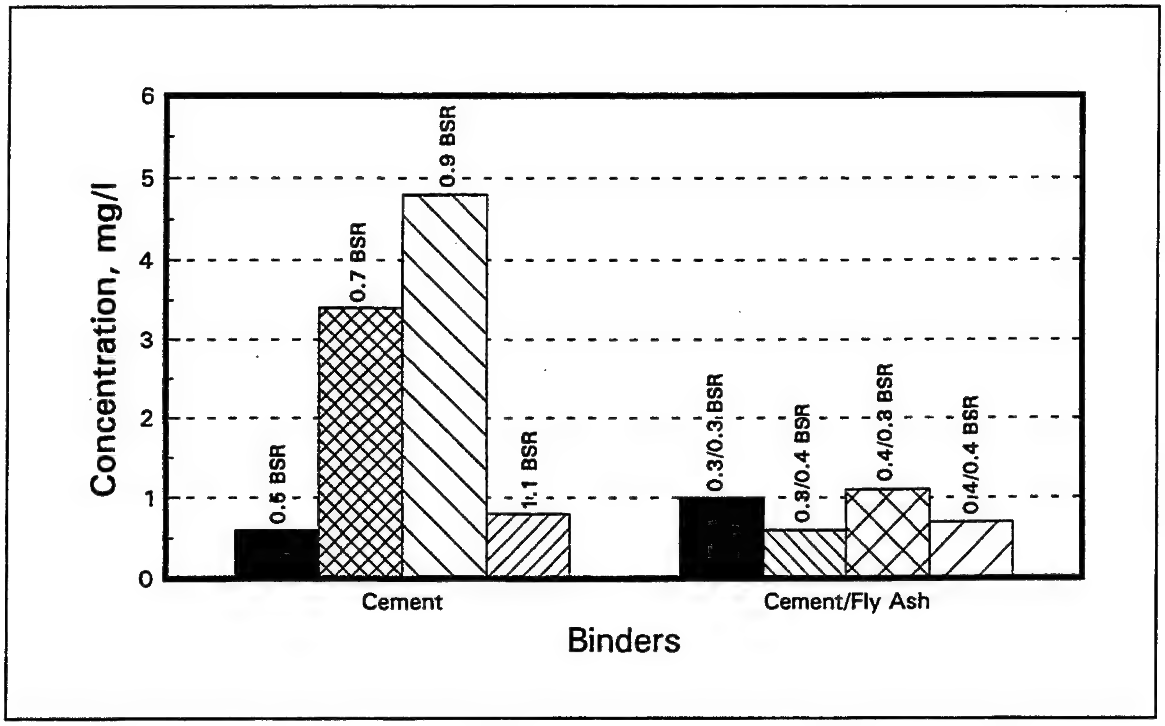


Figure 11. Average TRPH results of TCLP for detailed evaluation

Figure 12 presents the ratio of TRPH leached from the treated samples to the TRPH of the untreated sludge sample. The average TRPH concentration from the TCLP for the untreated sludge was 1,645 mg/ℓ; the average treated sludge TRPH concentration from the TCLP test was 1.6 mg/ℓ, a two order of magnitude reduction. Figure 12 shows that all of the BSRs used in the detailed evaluation portion of this study reduced the amount of TRPH leached in the TCLP by more than 99.6 percent. The cement and cement/fly ash BSRs performed well for the reduction of TRPHs leached during the TCLP test.

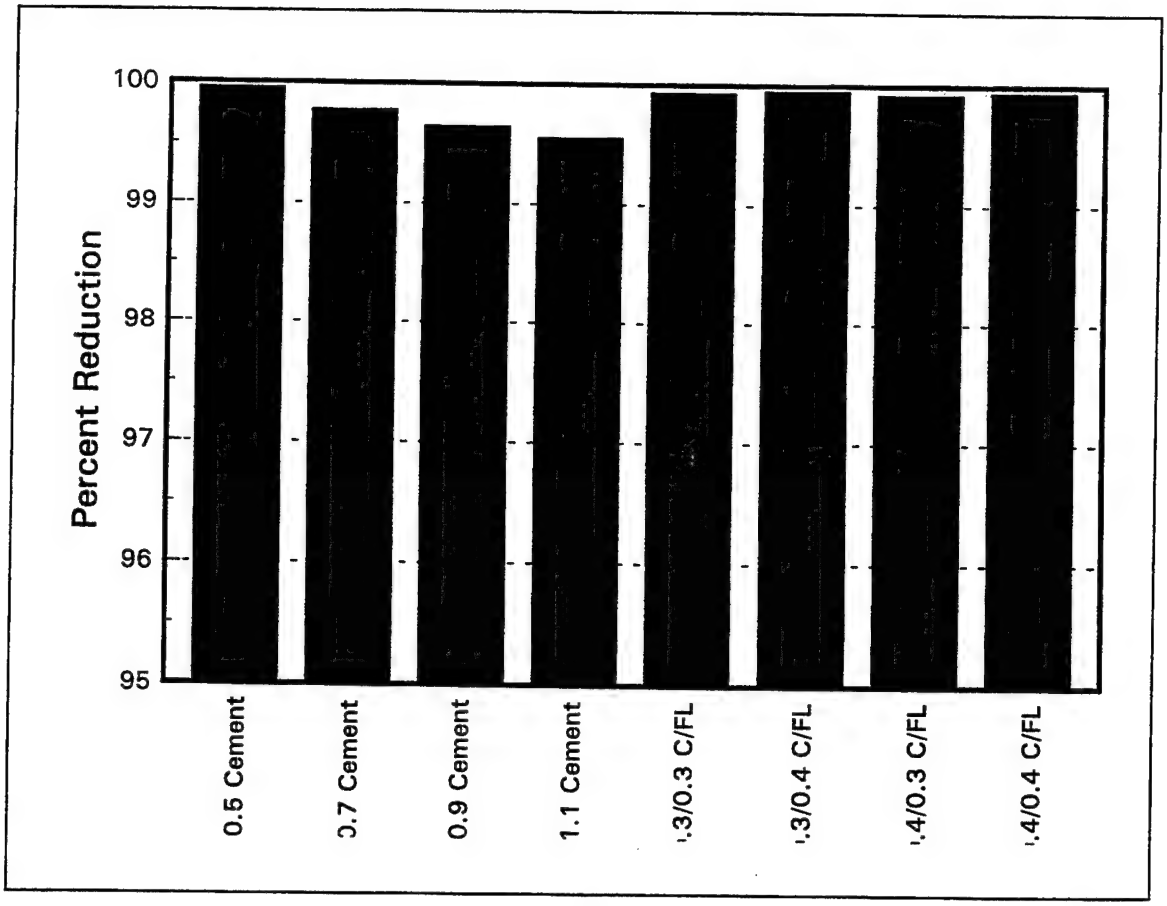


Figure 12. Average reduction of TRPHs leached from treated samples during TCLP as compared with the untreated sludge TCLP

# 4 Conclusions and Recommendations

## Conclusions

A laboratory study was conducted to investigate the effects of two S/S processes on a contaminated sludge from the washracks located at the Fort Polk Army Installation. This study indicated that the addition of 1.35 dicalcium silicate to oily sludge was needed for the success of the solidification process. Physical and chemical tests were performed on the solidified/stabilized specimens. Based on the results of these tests, the following conclusions can be made:

*a.* BSRs evaluated produced materials with UCSs well above the 50-psi criterion.

*b.* The addition of water to the sludge is not needed for the hydration of the binders to develop strength.

*c.* The S/S with additives sludge sets with 48 hr, and no free liquid was observed.

*d.* The S/S processing of the sludge was effective in reducing the mobility of TRPHs in the sludge.

*e.* Dicalcium silicate needs to be added to the sludge to absorb the petroleum hydrocarbons present in the sludge to strengthen development and prevent the formation of free water.

*f.* Dicalcium silicate is superior to organophilic clays for the S/S of oily sludge.

## Recommendations

Based on the results of this study, the binder ratio of 0.3/0.4 cement/fly ash with the addition of 1.35 dicalcium silicate is the process that best achieved the goals for the treatability study of oily washrack sludge. It is recommended for demonstration and/or pilot-scale development.

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# Appendix A
# Results of Physical and Chemical Tests Performed on the Untreated Washrack Sludge From Fort Polk Army Installation

**Table A1**
**Results of Physical Tests Performed on the Untreated Washrack Sludge**

| Sample ID | Moisture Content, Percent | Bulk Density $lb/ft^3$ | UCS, psi | Cone Index psi |
|---|---|---|---|---|
| A1 | 22 | 40.3 | 0 | 0 |
| A2 | 20 | 35.3 | 0 | 0 |
| A3 | 21 | 37.4 | 0 | 0 |
| B1 | 20 | 38.7 | 0 | 0 |
| B2 | 22 | 29.5 | 0 | 0 |
| B3 | 26 | 34.3 | 0 | 0 |

**Table A2**
**Chemical Analysis for the Untreated Washrack Sludge, mg/kg**

| ID | As | Ba | Cd | Cr | Pb | Hg | Se | Ag | TRPH |
|---|---|---|---|---|---|---|---|---|---|
| A | <0.20 | 14.5 | 0.65 | 0.79 | 2.57 | <0.100 | <0.20 | 0.10 | 314,000 |
| B | <0.20 | 13.1 | 0.81 | 0.98 | 4.02 | <0.100 | <0.20 | 0.10 | 314,000 |

**Table A3**
**TCLP Chemical Analysis for the Untreated Washrack Sludge, mg/ℓ**

| ID | As | Ba | Cd | Cr | Pb | Hg | Se | Ag | TRPH |
|---|---|---|---|---|---|---|---|---|---|
| A | <0.002 | 0.182 | 0.0005 | <0.001 | 0.012 | <0.0002 | <0.002 | <0.010 | 2,040 |
| B | <0.002 | 0.196 | 0.0004 | 0.001 | 0.128 | <0.0002 | <0.002 | <0.010 | 1,250 |

# Appendix B
# Results of Initial Screening Test for Washrack Sludge

**Table B1**
**Cone Index for IST Cement Binder**

| Cement BSR | Replicate | Cone Index, psi | | | | |
|---|---|---|---|---|---|---|
| | | 2 hr | 4 hr | 8 hr | 24 hr | 48 hr |
| 0.8 | A | 0 | 0 | 0 | 0 | 0 |
| 0.8 | B | 0 | 0 | 0 | 0 | 0 |
| 1.5 | A | 0 | 0 | 15 | 10 | 10 |
| 1.5 | B | 0 | 0 | 20 | 10 | 10 |
| 2.0 | A | 0 | 10 | 10 | 10 | 15 |
| 2.0 | B | 0 | 10 | 10 | 10 | 25 |
| 3.0 | A | 10 | 15 | 25 | 70 | 80 |
| 3.0 | B | 10 | 20 | 20 | 70 | 100 |

**Table B2**
**Cone Index for IST Cement Binder With Klensorb Organophilic Clay Absorbent**

| Cement BSR | Klensorb BSR | Replicate | Cone Index, psi | | | | |
|---|---|---|---|---|---|---|---|
| | | | 2 hr | 4 hr | 8 hr | 24 hr | 48 hr |
| 1.0 | 0.5 | A | 0 | 0 | 0 | 0 | 20 |
| 1.0 | 0.5 | B | 0 | 0 | 0 | 0 | 20 |
| 1.0 | 1.2 | A | 0 | 20 | 25 | 50 | 80 |
| 1.0 | 1.2 | B | 0 | 20 | 30 | 60 | 100 |
| 1.0 | 1.8 | A | 0 | 0 | 0 | 60 | 190 |
| 1.0 | 1.8 | B | 0 | 0 | 0 | 60 | 210 |

## Table B3
## Cone Index for IST Cement Binder With Dicalcium Silicate Absorbent

| Cement BSR | Dicalcium Silicate BSR | Replicate | Cone Index, psi | | | | |
|---|---|---|---|---|---|---|---|
| | | | 2 hr | 4 hr | 8 hr | 24 hr | 48 hr |
| 1.0 | 0.5 | A | 0 | 0 | 10 | 25 | 80 |
| 1.0 | 0.5 | B | 0 | 0 | 25 | 25 | 90 |
| 1.0 | 1.0 | A | 0 | 0 | 25 | 270 | 750 |
| 1.0 | 1.0 | B | 0 | 0 | 40 | 290 | 750 |
| 1.0 | 1.5 | A | 180 | 260 | 750 | 750 | 750 |
| 1.0 | 1.5 | B | 180 | 270 | 750 | 750 | 750 |
| 1.0 | 2.0 | A | 0[1] | 0[1] | 0[1] | 0[1] | 0[1] |
| 1.0 | 2.0 | B | 0[1] | 0[1] | 0[1] | 0[1] | 0[1] |

[1] Sample crumbled during test.

# Appendix C
# Results of Physical and Chemical Tests Performed on Samples Prepared for the Detailed Evaluation

**Table C1**
**Results of Moisture and 28-Day Bulk Density for Detailed Evaluation Samples**

| BSR | Replicate | Moisture Content Percent | 28-Day Bulk Density, lb/ft$^3$ |
|---|---|---|---|
| **Cement Binder** | | | |
| 0.5 | A | 22.1 | 93.56 |
| 0.5 | B | 22.6 | 92.30 |
| 0.7 | A | 20.0 | 90.24 |
| 0.7 | B | 21.8 | 92.41 |
| 0.9 | A | 18.0 | 102.37 |
| 0.9 | B | 18.0 | 101.51 |
| 1.1 | A | 14.1 | 102.38 |
| 1.1 | B | 14.0 | 102.83 |
| **Cement/Fly Ash Binder** | | | |
| 0.3/0.3 | A | 24.0 | 86.54 |
| 0.3/0.3 | B | 23.0 | 89.39 |
| 0.3/0.4 | A | 22.2 | 55.04 |
| 0.3/0.4 | B | 22.4 | 54.42 |
| 0.4/0.3 | A | 20.9 | 51.21 |
| 0.4/0.3 | B | 20.5 | 55.62 |
| 0.4/0.4 | A | 21.5 | 45.61 |
| 0.4/0.4 | B | 21.5 | 53.70 |

**Table C2**
**Results of CI for Samples Prepared for the Detailed Evaluation**

| BSR | Replicate | Cone Index, psi | | | | |
|---|---|---|---|---|---|---|
| | | 2 hr | 4 hr | 8 hr | 24 hr | 48 hr |
| **Cement Binder** | | | | | | |
| 0.5 | A | 0 | 0 | 56 | 633 | 750 |
| 0.5 | B | 0 | 0 | 116 | 600 | 750 |
| 0.7 | A | 0 | 143 | 206 | 650 | 750 |
| 0.7 | B | 0 | 126 | 233 | 616 | 750 |
| 0.9 | A | 133 | 616 | 750 | 750 | 750 |
| 0.9 | B | 136 | 683 | 750 | 750 | 750 |
| 1.1 | A | 700 | 750 | 750 | 750 | 750 |
| 1.1 | B | 716 | 750 | 750 | 750 | 750 |
| **Cement/Fly Ash Binder** | | | | | | |
| 0.3/0.3 | A | 0 | 28 | 126 | 475 | 566 |
| 0.3/0.3 | B | 0 | 31 | 158 | 460 | 500 |
| 0.3/0.4 | A | 45 | 103 | 146 | 583 | 750 |
| 0.3/0.4 | B | 42 | 102 | 226 | 716 | 750 |
| 0.4/0.3 | A | 43 | 113 | 226 | 716 | 750 |
| 0.4/0.3 | B | 43 | 113 | 266 | 716 | 750 |
| 0.4/0.4 | A | 90 | 146 | 375 | 750 | 750 |
| 0.4/0.4 | B | 63 | 153 | 433 | 750 | 750 |

**Table C3**
**Results of UCS for Samples Prepared for Detailed Evaluation**

| BSR | Replicate | Cure Time | UCS, psi |
|---|---|---|---|
| | | **Cement Binder** | |
| 0.5 | A1 | 7 | 182 |
| 0.5 | A2 | 7 | 185 |
| 0.5 | B1 | 7 | 199 |
| 0.5 | B2 | 7 | 197 |
| 0.5 | A1 | 14 | 223 |
| 0.5 | A2 | 14 | 225 |
| 0.5 | B1 | 14 | 229 |
| 0.5 | B2 | 14 | 231 |
| 0.5 | A1 | 21 | 238 |
| 0.5 | A2 | 21 | 229 |
| 0.5 | B1 | 21 | 229 |
| 0.5 | B2 | 21 | 234 |
| 0.5 | A1 | 28 | 301 |
| 0.5 | A2 | 28 | 293 |
| 0.5 | B1 | 28 | 230 |
| 0.5 | B2 | 28 | 242 |
| 0.7 | A1 | 7 | 289 |
| 0.7 | A2 | 7 | 283 |
| 0.7 | B1 | 7 | 273 |
| 0.7 | B2 | 7 | 281 |
| 0.7 | A1 | 14 | 342 |
| 0.7 | A2 | 14 | 277 |
| 0.7 | B1 | 14 | 337 |
| 0.7 | B2 | 14 | 283 |
| 0.7 | A1 | 21 | 355 |
| 0.7 | A2 | 21 | 356 |
| 0.7 | B1 | 21 | 257 |
| 0.7 | B2 | 21 | 260 |
| 0.7 | A1 | 28 | 349 |
| 0.7 | A2 | 28 | 343 |

*(Sheet 1 of 5)*

| Table C3 (Continued) | | | |
|---|---|---|---|
| **BSR** | **Replicate** | **Cure Time** | **UCS, psi** |
| **Cement Binder** | | | |
| 0.7 | B1 | 28 | 299 |
| 0.7 | B2 | 28 | 294 |
| 0.9 | A1 | 7 | 536 |
| 0.9 | A2 | 7 | 534 |
| 0.9 | B1 | 7 | 676 |
| 0.9 | B2 | 7 | 680 |
| 0.9 | A1 | 14 | 1,001 |
| 0.9 | A2 | 14 | 996 |
| 0.9 | B1 | 14 | 628 |
| 0.9 | B2 | 14 | 639 |
| 0.9 | A1 | 21 | 794 |
| 0.9 | A2 | 21 | 786 |
| 0.9 | B1 | 21 | 921 |
| 0.9 | B2 | 21 | 927 |
| 0.9 | A1 | 28 | 1,035 |
| 0.9 | A2 | 28 | 1,037 |
| 0.9 | B1 | 28 | 651 |
| 0.9 | B2 | 28 | 655 |
| 1.1 | A1 | 7 | 772 |
| 1.1 | A2 | 7 | 735 |
| 1.1 | B1 | 7 | 731 |
| 1.1 | B2 | 7 | 770 |
| 1.1 | A1 | 14 | 873 |
| 1.1 | A2 | 14 | 868 |
| 1.1 | B1 | 14 | 412 |
| 1.1 | B2 | 14 | 420 |
| 1.1 | A1 | 21 | 1,002 |
| 1.1 | A2 | 21 | 1,007 |
| 1.1 | B1 | 21 | 231 |
| 1.1 | B2 | 21 | 238 |

*(Sheet 2 of 5)*

**Table C3 (Continued)**

| BSR | Replicate | Cure Time | UCS, psi |
|---|---|---|---|
| **Cement Binder** | | | |
| 1.1 | A1 | 28 | 752 |
| 1.1 | A2 | 28 | 743 |
| 1.1 | B1 | 28 | 743 |
| 1.1 | B2 | 28 | 746 |
| **Cement/Fly Ash Binder** | | | |
| 0.3/0.3 | A1 | 7 | 349 |
| 0.3/0.3 | A2 | 7 | 361 |
| 0.3/0.3 | B1 | 7 | 341 |
| 0.3/0.3 | B2 | 7 | 334 |
| 0.3/0.3 | A1 | 14 | 374 |
| 0.3/0.3 | A2 | 14 | 370 |
| 0.3/0.3 | B1 | 14 | 205 |
| 0.3/0.3 | B2 | 14 | 207 |
| 0.3/0.3 | A1 | 21 | 256 |
| 0.3/0.3 | A2 | 21 | 253 |
| 0.3/0.3 | B1 | 21 | 343 |
| 0.3/0.3 | B2 | 21 | 350 |
| 0.3/0.3 | A1 | 28 | 326 |
| 0.3/0.3 | A2 | 28 | 315 |
| 0.3/0.3 | B1 | 28 | 340 |
| 0.3/0.3 | B2 | 28 | 343 |
| 0.3/0.4 | A1 | 7 | 293 |
| 0.3/0.4 | A2 | 7 | 300 |
| 0.3/0.4 | B1 | 7 | 289 |
| 0.3/0.4 | B2 | 7 | 297 |
| 0.3/0.4 | A1 | 14 | 363 |
| 0.3/0.4 | A2 | 14 | 371 |
| 0.3/0.4 | B1 | 14 | 339 |
| 0.3/0.4 | B2 | 14 | 328 |
| 0.3/0.4 | A1 | 21 | 394 |

*(Sheet 3 of 5)*

**Table C3 (Continued)**

| BSR | Replicate | Cure Time | UCS, psi |
|---|---|---|---|
| **Cement/Fly Ash Binder** | | | |
| 0.3/0.4 | A2 | 21 | 401 |
| 0.3/0.4 | B1 | 21 | 322 |
| 0.3/0.4 | B2 | 21 | 330 |
| 0.3/0.4 | A1 | 28 | 562 |
| 0.3/0.4 | A2 | 28 | 560 |
| 0.3/0.4 | B1 | 28 | 482 |
| 0.3/0.4 | B2 | 28 | 485 |
| 0.4/0.3 | A1 | 7 | 203 |
| 0.4/0.3 | A2 | 7 | 224 |
| 0.4/0.3 | B1 | 7 | 268 |
| 0.4/0.3 | B2 | 7 | 271 |
| 0.4/0.3 | A1 | 14 | 392 |
| 0.4/0.3 | A2 | 14 | 381 |
| 0.4/0.3 | B1 | 14 | 310 |
| 0.4/0.3 | B2 | 14 | 321 |
| 0.4/0.3 | A1 | 21 | 388 |
| 0.4/0.3 | A2 | 21 | 366 |
| 0.4/0.3 | B1 | 21 | 396 |
| 0.4/0.3 | B2 | 21 | 417 |
| 0.4/0.3 | A1 | 28 | 396 |
| 0.4/0.3 | A2 | 28 | 389 |
| 0.4/0.3 | B1 | 28 | 431 |
| 0.4/0.3 | B2 | 28 | 448 |
| 0.4/0.4 | A1 | 7 | 325 |
| 0.4/0.4 | A2 | 7 | 347 |
| 0.4/0.4 | B1 | 7 | 315 |
| 0.4/0.4 | B2 | 7 | 330 |
| 0.4/0.4 | A1 | 14 | 350 |
| 0.4/0.4 | A2 | 14 | 349 |
| 0.4/0.4 | B1 | 14 | 294 |

*(Sheet 4 of 5)*

| Table C3 (Concluded) | | | |
|---|---|---|---|
| BSR | Replicate | Cure Time | UCS, psi |
| Cement/Fly Ash Binder | | | |
| 0.4/0.4 | B2 | 14 | 305 |
| 0.4/0.4 | A1 | 21 | 424 |
| 0.4/0.4 | A2 | 21 | 426 |
| 0.4/0.4 | B1 | 21 | 348 |
| 0.4/0.4 | B2 | 21 | 346 |
| 0.4/0.4 | A1 | 28 | 550 |
| 0.4/0.4 | A2 | 28 | 488 |
| 0.4/0.4 | B1 | 28 | 332 |
| 0.4/0.4 | B2 | 28 | 425 |
| | | | *(Sheet 5 of 5)* |

**Table C4**
**Metal Concentration of TCLP Samples for the Detailed Evaluation, mg/ℓ**

| BSR | Replicate | As | Ba | Cd | Cr | Pb | Hg | Se | Ag |
|---|---|---|---|---|---|---|---|---|---|
| Cement Binder | | | | | | | | | |
| 0.5 | A | <0.002 | 0.587 | <0.0002 | 0.0364 | <0.002 | <0.002 | 0.003 | <0.01 |
| 0.5 | B | 0.003 | 0.747 | <0.0002 | 0.0422 | <0.002 | <0.002 | 0.003 | <0.01 |
| 0.7 | A | 0.003 | 1.12 | <0.0002 | 0.0436 | 0.002 | <0.002 | <0.002 | <0.01 |
| 0.7 | B | <0.002 | 1.23 | <0.0002 | 0.0316 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.9 | A | 0.003 | 1.48 | <0.0002 | 0.0258 | 0.002 | <0.002 | <0.002 | <0.01 |
| 0.9 | B | 0.004 | 1.42 | <0.0002 | 0.0298 | 0.005 | <0.002 | <0.002 | <0.01 |
| 1.1 | A | 0.003 | 1.45 | <0.0002 | 0.0320 | <0.002 | <0.002 | <0.002 | <0.01 |
| 1.1 | B | <0.002 | 1.49 | <0.0002 | 0.0329 | <0.002 | <0.002 | <0.002 | <0.01 |
| Cement/Fly Ash Binder | | | | | | | | | |
| 0.3/0.3 | A | <0.002 | 1.36 | <0.0002 | 0.0315 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.3/0.3 | B | <0.002 | 1.46 | <0.0002 | 0.0311 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.3/0.4 | A | <0.002 | 1.25 | <0.0002 | 0.0385 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.3/0.4 | B | <0.002 | 1.14 | <0.0002 | 0.0377 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.3 | A | <0.002 | 1.21 | <0.0002 | 0.0294 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.3 | B | <0.002 | 1.36 | <0.0002 | 0.0266 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.4 | A | <0.002 | 1.22 | <0.0002 | 0.0308 | <0.002 | <0.002 | <0.002 | <0.01 |
| 0.4/0.4 | B | <0.002 | 1.19 | <0.0002 | 0.0299 | <0.002 | <0.002 | <0.002 | <0.01 |

**Table C5**
**TRPH Concentration for TCLP Samples for the Detailed Evaluation**

| BSR | Replicate | Total Recoverable Petroleum Hydrocarbons, mg/ℓ |
|---|---|---|
| **Cement Binder** | | |
| 0.5 | A | 0.7 |
| 0.5 | B | <0.5 |
| 0.7 | A | 4.5 |
| 0.7 | B | 2.5 |
| 0.9 | A | 4.7 |
| 0.9 | B | 6.7 |
| 1.1 | A | 0.8 |
| 1.1 | B | 0.4[1] |
| **Cement/Fly Ash Binder** | | |
| 0.3/0.3 | A | 0.9 |
| 0.3/0.3 | B | 1.0 |
| 0.3/0.4 | A | 0.9 |
| 0.3/0.4 | B | 0.3[1] |
| 0.4/0.3 | A | 1.8 |
| 0.4/0.3 | B | <0.5 |
| 0.4/0.4 | A | 0.9 |
| 0.4/0.4 | B | 0.7 |

[1] Detection of the analyte; however, uncertainty exists due to the limits of the available analytic method.

# REPORT DOCUMENTATION PAGE

*Form Approved*
*OMB No. 0704-0188*



**1. AGENCY USE ONLY** *(Leave blank)*

**2. REPORT DATE**
September 1996

**3. REPORT TYPE AND DATES COVERED**
Final report

**4. TITLE AND SUBTITLE**
An Evaluation of Solidification/Stabilization for Treatment of a Petroleum Hydrocarbon Contaminated Sludge From Fort Polk Army Installation, Louisiana

**5. FUNDING NUMBERS**

**6. AUTHOR(S)**
Michael G. Channell, Kurt T. Preston

**7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)**
U.S. Army Engineer Waterways Experiment Station
3909 Halls Ferry Road, Vicksburg, MS 39180-6199

**8. PERFORMING ORGANIZATION REPORT NUMBER**
Miscellaneous Paper EL-96-8

**9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)**
U.S. Army Installation, Fort Polk
Fort Polk, LA 71459-7100

**10. SPONSORING/MONITORING AGENCY REPORT NUMBER**

**11. SUPPLEMENTARY NOTES**
Available from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

**12a. DISTRIBUTION/AVAILABILITY STATEMENT**
Approved for public release; distribution is unlimited.

**12b. DISTRIBUTION CODE**

**13. ABSTRACT** *(Maximum 200 words)*

In the course of normal operations and training, soldiers and civilian personnel operate many Army vehicles on a day-to-day basis. These vehicles must be cleaned before they can be returned to the motor pool area of an Army base. The cleaning of these vehicles has posed a problem with the operation and maintenance of oil/water separators located at vehicle washrack facilities. An oily sludge forms in the oil/water separator and is hard to handle and cannot be disposed of in an ordinary manner. This study used solidification/stabilization to treat the oily sludge found in the vehicle washrack oil/water separators.

Solidification/stabilization is usually used to treat soils and sludges that contain heavy metals. Organic compounds, such as petroleum hydrocarbons found in the sludge, interfere with the setting of the solidification binding materials and thus produce a material that is not desirable for a treatment alternative. This study incorporates the use of dicalcium silicate as an additive to the solidification process to increase the strength and reduce the leachability of the petroleum hydrocarbons found in the sludge. This study shows that dicalcium silicate improves the handling characteristics of the sludge and reduces the leachability of the contaminants from the washrack sludge.

**14. SUBJECT TERMS**

| | |
|---|---|
| Dicalcium silicate | Solidification |
| Leachability | Stabilization |
| Petroleum hydrocarbons | Washrack |
| Sludge | |

**15. NUMBER OF PAGES**
47

**16. PRICE CODE**

| 17. SECURITY CLASSIFICATION OF REPORT | 18. SECURITY CLASSIFICATION OF THIS PAGE | 19. SECURITY CLASSIFICATION OF ABSTRACT | 20. LIMITATION OF ABSTRACT |
|---|---|---|---|
| UNCLASSIFIED | UNCLASSIFIED | | |